

UNITED STATES PATENT OFFICE

2,121,138

METHOD FOR MANUFACTURING SMOKE-
LESS POWDER

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No Drawing. Application January 11, 1937,
Serial No. 120,043

12 Claims. (Cl. 52—22)

This invention relates to a method for the manufacture of smokeless powder and more particularly to the manufacture of the so-called double base powder comprising nitrocellulose and nitroglycerine, nitroglycol, or the like.

Heretofore, it has been known to produce smokeless powder by incorporating a liquid high explosive such as nitro-glycerine, nitroglycol or the like with nitrocellulose for the production of a colloid and then dividing this colloid into grains of the desired shape and size by pressing and granulating operations or into flakes by rolling and cutting operations. Such powder is known to the art and has included other constituents adopted to maintain its stability, vary its burning rate, etc.

In the production of such double base powders it has been customary to admix alcohol wet nitrocellulose, nitroglycerine or other liquid high explosives and a solvent such as acetone to produce a colloid. This colloid is then grained and the alcohol and acetone eliminated from the grains.

The procedures of the prior art are recognized as unsatisfactory due to the danger arising from the inflammability of the alcohol and solvents for nitroglycerine or the like and the expense involved in their recovery.

Heretofore, it has also been known to eliminate the objections to the use of a volatile solvent by admixing the nitrocellulose with water in the form of a slurry or pulp, adding to this nitroglycerine or the like by pouring the nitroglycerine or the like into the nitrocellulose slurry and agitating the mixture until all of the nitroglycerine is absorbed by the nitrocellulose. The water is then removed and the mixture colloided by the application of heat and pressure either on rolls or in a press.

It is recognized that such a procedure will fail to give a uniform distribution of the nitroglycerine on or in the nitrocellulose. Furthermore, the excess water must be removed by pressing or centrifuging. This water carries with it a small amount of nitroglycerine which represents an economic loss as well as a hazard in the disposal of the water.

A modification of this procedure is described by Lundholm et al. in British Patent No. 10,376, issued in 1889. According to this procedure the nitrocellulose may be made into a slurry or pulp with water and finely divided nitroglycerine added. The dispersion of the nitroglycerine may be accomplished by the use of a spray or jet in which case it is added directly to the nitrocotton

pulp or it may be accomplished by first emulsifying the nitroglycerine in water. In this procedure, as in the previous one, it is necessary to eliminate excess water.

In addition to the disadvantage of the excess water which must be removed, Lundholm et al. have the hazard of dispersing nitroglycerine by means of a spray or the difficulty of emulsifying nitroglycerine in water by agitation. Not only must very violent agitation be required to emulsify the nitroglycerine, but the emulsion formed will be very unstable and it is necessary to maintain the agitation until all of the nitroglycerine is taken up by the nitrocotton. At any time when vigorous agitation of the Lundholm et al. emulsion ceases, the nitroglycerine separates from the water and becomes as sensitive to shock, as if the water were not present.

In the above procedures it is advisable to age or cure the powder, preferably at elevated temperatures, to assure that the nitroglycerine is entirely taken into the cotton fiber. If any free nitroglycerine remains, the mixture will be very hazardous to roll or press. This operation adds to the hazard of manufacture in that it requires additional handling of the explosive mixture.

Now, in accordance with my invention, there is provided a method for combining a liquid high explosive, as, for example, nitroglycerine, nitroglycol or the like and nitrocellulose in the production of smokeless powder without the disadvantages of the old methods.

The procedure in accordance with this invention is based on the surprising discovery that a stable emulsion of nitroglycerine, nitroglycol or the like in dispersed phase in water, when admixed with water wet nitrocellulose will produce a colloid which is almost non-inflammable and non-explosive until after the water is largely removed. This discovery makes possible a great reduction in the hazard and the time required in the manufacture of smokeless powder which is economically advantageous. Furthermore, it insures a uniform distribution of the nitroglycerine, nitroglycol or the like in the powder. In marked contrast to straight nitroglycerine, a stable emulsion of nitroglycerine is very insensitive to shock and cannot be detonated with a blasting cap.

The method in accordance with this invention will be efficient and economical and will be adaptable for the production of smokeless powder including a high explosive, as nitroglycerin, nitroglycol, or the like, and also including constituents adapted to promote the stability, to control burning rate, etc., of the powder, and/or

including a high-boiling solvent or plasticizer, as dibutyl phthalate, tricresyl phosphate, or the like.

It is also known (U. S. Patent 1,999,828) to treat nitrocellulose gunpowder for the production of a double base powder by mixing finely ground nitrocellulose powder with a stable emulsion of nitroglycerine, allowing the mixture to stand, spreading out in trays and drying, thus allowing the water gradually to evaporate from the emulsion, leaving on the surface of the grains of powder the nitroglycerine, which will gradually penetrate the grains and come to an equilibrium throughout the grains, the relatively small amount of nitroglycerine left on the surface of the grains being too small to exert any appreciable effect on the ballistics of the double base powder so produced.

Generally speaking, the method in accordance with this invention will comprise the admixture of nitrocellulose wet with water with a stable aqueous emulsion of a high explosive, or a mixture of high explosives, as nitroglycerine, or equivalent, as nitroglycol, for the formation of a colloid, graining the colloid, and effecting final elimination of water by evaporation.

In proceeding in accordance with this invention, constituents for promoting the stability of the powder, for controlling its burning rate, or the like, may be incorporated before, during, or after the admixing of the nitrocellulose and aqueous emulsion of nitroglycerine, or the like, or after the graining of the powder and the elimination of water, as desire and good practice may dictate. Likewise, if desired, an additional quantity of nitrocellulose may be incorporated with the colloid prior to its final colloid, and added nitrocellulose may be of the same or of a different nitrogen content with respect to the nitrocellulose used to form the colloid.

In proceeding, any suitable aqueous emulsion of, for example, nitroglycerine or the like, of the oil-in-water type, formed with the use of any suitable emulsifying agent, may be used. Thus, for example, an emulsion comprising nitroglycerine or the like, emulsified in water using as an emulsifying agent a water-soluble hydrophilic colloid, such as, for example, agar-agar, gum tragacanth, gelatine, etc.; water-soluble carbohydrate ethers, as methyl cellulose, low-substituted, water-soluble ethyl cellulose, water-soluble glycol cellulose, etc. will be found satisfactory. Ordinarily, I prefer to use water-soluble methyl cellulose as the emulsifying agent, since it is an excellent emulsifying agent and does not decompose or become rancid on storage. The emulsification of the nitroglycerine or its equivalent may be accomplished in any well known manner, as by addition of the nitroglycerine to water containing the emulsifying agent with agitation. A suitable emulsion for use in accordance with this invention will comprise a liquid high explosive, as, for example, nitroglycerine, nitroglycol, or the like, emulsified in disperse phase in water containing dissolved therein a water-soluble hydrophilic colloid. The amount of the liquid high explosive contained therein may vary over a comparatively wide range, as, for example, from about 25% to about 95% by weight of the finished emulsion. Ordinarily, I prefer to use an emulsion containing a liquid high explosive in amount within the range of about 60% to about 85% by weight of the finished emulsion. The aqueous phase of the emulsion may contain from

about 0.2% to about 10.0% by weight of the water-soluble hydrophilic colloid.

As illustrative of practical adaptation of the method in accordance with this invention, for example, 1617 grams of 13.2% nitrogen nitro-cotton containing 25.0% of water is mixed in any usual powder mixer, such as a Werner-Pfleiderer mixer, with 1242 grams of an emulsion comprising 974 grams of nitroglycerine, 192 grams of water and 8 grams of methyl cellulose for 5 to 30 minutes at atmospheric temperature. If desired, a stabilizer, as diphenylamine, diethyldiphenyl urea, or the like, a suitable deterrent, a suitable plasticizer, or the like as commonly used in the art may be dissolved in the nitroglycerine before emulsification, or if liquid, emulsified with the nitroglycerine or otherwise added to the system.

The water-wet mass resultant from the mixing having a physical structure intermediate between that of the original and a semi-colloided nitrocellulose, will be formed into a coherent, colloided mass by pressure applied by passing between rolls or in a hydraulic press. The colloid action of the nitroglycerine will be accelerated if pressure is applied at a temperature of about 50° C. to about 100° C. The coherent colloided mass is divided into pieces or grains of desired shape and size by cutting or other well known means for graining colloided powder. The water may be removed from the plastic during the rolling operation, or it may be removed from the finished grains, as may be desired.

The dried powder grains may be subjected to any further treatment such as coating, glazing, or the like, as may be desired.

If desired, the semi-colloided mass, before graining, may be combined with additional nitrocellulose of the same or of higher or of lower nitrogen content. Thus, the colloided mass may be combined with, for example, further nitrocellulose, of about 13.25% nitrogen, or of about 11.0% nitrogen, wet with 30.0% water, more or less, by incorporation on and passing through rolls.

As further illustrative of practical adaptation of the invention, an aqueous emulsion consisting of 1043 grams of nitroglycerine, 240 grams of water, and 10 grams of methyl cellulose is mixed with 1226 grams of 12.6% nitrogen nitrocotton containing 20.20% water and 22.6 grams of symmetrical diethyldiphenyl urea.

Ethylene glycol dinitrate, diethylene glycol dinitrate, or other liquid high explosives may be substituted for the nitroglycerine in the above powders.

In proceeding in accordance with this invention, customary hazards in the handling and transportation of the nitroglycerine will be eliminated by forming an emulsion as soon as the nitroglycerine is ready to be used. The emulsion is stable and after preparation may be kept indefinitely without agitation until desired for use. By use of such an emulsion, the use of excess water can be eliminated without decreasing the safety of the process.

Uniform distribution of the nitroglycerine on the nitrocellulose can be readily obtained, and since the emulsified nitroglycerine is insensitive to shock and the emulsion is stable until the finely dispersed particles of nitroglycerine are absorbed into the nitrocotton fiber, we can hasten the absorption of the nitroglycerine and the final colloid of the powder by applying heat and pressure to the mass immediately after the emulsion and nitrocotton are mixed together, without the

hazard of the probable presence of free nitroglycerine encountered in the processes of the prior art.

In carrying out the method according to this invention, it will be found that the emulsion and nitrocellulose will combine readily in the mixing operation and that upon the application of pressure a coherent, colloided mass will be readily formed.

It will be clearly understood that this invention from the broad standpoint is not dependent upon the use of any particular liquid high explosive or mixture and/or solutions of high explosives, or upon any particular concentration of high explosive in the emulsion, or the use of any particular emulsifying agent or any particular procedure for emulsifying the high explosive. Broadly, this invention contemplates the application of any emulsified high explosive, processed with nitrocellulose and with or without other explosive or non-explosive modifiers, to produce a propellant of controlled rate of burning or detonation.

What I claim and desire to protect by Letters Patent is:

1. The method of producing dense, colloided smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing a water-soluble hydrophilic colloid in solution.

2. The method of producing dense, colloided smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing a water-soluble carbohydrate ether in solution.

3. The method of producing dense, colloided smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing methyl cellulose in solution.

4. The method of producing dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing water-soluble ethyl cellulose.

5. The method of producing dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing water-soluble glycol cellulose.

6. The method of producing dense, colloided,

smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing a water-soluble carbohydrate ether, and subjecting the mixture to pressure to form a coherent colloid.

7. The method of producing dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water with nitroglycerine emulsified in water containing a water-soluble carbohydrate ether, subjecting the mixture to pressure to form a coherent colloid, grain- ing the resultant colloid, and drying water from the grains.

8. The method of producing a dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water with modifying agents adapted to control the burning rate of the smokeless powder and with nitroglycerine emulsified in water containing a water soluble carbohydrate ether in solution.

9. The method of producing a dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water with modifying agents adapted to control the burning rate of the smokeless powder and nitroglycerine and liquid deterrent emulsified in water containing a water-soluble carbohydrate ether in solution.

10. The method of producing a dense, colloided, smokeless powder which includes admixing fibrous nitrocellulose wet with water and a nitrated polyhydric alcohol emulsified in water containing a water-soluble carbohydrate ether in solution.

11. The method of producing dense, colloided smokeless powder which includes admixing fibrous nitrocellulose wet with water and containing from about 12.6% to about 13.25% of nitrogen, with nitroglycerine emulsified in water containing a water-soluble, hydrophilic colloid in solution.

12. The method of producing a dense, colloided smokeless powder which includes admixing fibrous nitrocellulose containing from about 12.6% to about 13.25% nitrogen, said nitrocellulose being wet with from about 20.20% to about 25% of water, with nitroglycerine emulsified in water containing a water-soluble, hydrophilic colloid in solution.

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